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Selective and Responsive Nanopore-Filled Membranes

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206023

6. AUTHORS
Yossef A. Elabd, Giuseppe R. Palmese

5d. PROJECT NUMBER

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14. ABSTRACT
The overall goal of this project is to develop a new class of breathable barrier membrane (i.e., selective for water vapor over harmful agents) with stimuli-responsive capabilities and the mechanical strength required for protective clothing for chemical and biological defense. These new materials are based on polymer-polymer nanocomposites of hydrophilic ionic polymer gels within a hydrophobic polymer host matrix. The specific tasks of this project include (1) synthesizing stimuli-responsive selective membranes based on polymer-polymer nanocomposites, (2)

15. SUBJECT TERMS
chem/bio protective clothing, membranes, nanoporous polymers, nanofibers, composites

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a. REPORT	b. ABSTRACT	c. THIS PAGE			Yossef Elabd
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Report Title

Selective and Responsive Nanopore-Filled Membranes

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A recent result from this work of interest is the fabrication and super conducting properties of Nafion nanofibers, which was recently published in a high-impact journal Nano Letters. In this paper, we reported the high proton conductivity of single high purity Nafion nanofiber (1.5 S/cm), which is an order of magnitude higher than the bulk Nafion film (~ 0.1 S/cm). We also observed a nanosize effect, where proton conductivity increases sharply with decreasing fiber diameter. X-ray scattering provides a rationale for these findings, where an oriented ionic morphology was observed in the nanofiber in contrast to the isotropic morphology in the bulk film. This work also demonstrates the successful fabrication of high purity Nafion nanofibers (~ 99.9 wt%) via electrospinning and higher humidity sensitivity for nanofibers compared to the bulk. These results should have a significant impact on not only the development of breathable barrier membranes, but also other applications such as fuel cells and sensors.

List of papers submitted or published that acknowledge ARO support during this reporting period. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

1. Dong, B.; Gwee, L.; Salas-de la Cruz, D.; Winey, K.I. Elabd, Y.A. Super Proton Conductive High Purity Nafion Nanofibers. Nano Letters 2010, 10, 3785-3790.

2. Rahmathullah, M. Aflal M.; Snyder, J.D.; Elabd, Y.A.; Palmese G.R. Nanoporous and Proton Conductive Hydrophobic-Hydrophilic Copolymer Thermoset Membranes. J. Polym. Sci. Pol. Phys. 2010, 48 (12), 1245-1255.

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9. Chen, H.; Palmese, G.R.; Elabd, Y.A. Membranes with Oriented Polyelectrolyte Nanodomains. Chem. Mater. 2006, 18, 4875-4881.

Number of Papers published in peer-reviewed journals: 9.00

(b) Papers published in non-peer-reviewed journals or in conference proceedings (N/A for none)

Number of Papers published in non peer-reviewed journals: 0.00

(c) Presentations

1. Chen, H.; Snyder, J.D.; Elabd, Y.A. Electrospinning Nafion Nanofibers. Annual Meeting of the American Institute of Chemical Engineers, Salt Lake City, UT, November 2007.

2. Rahmathullah, A.M.; Elabd Y.A.; Palmese, G.R. Design of Nanoporous, Proton-Conductive Polymer Thermosets. Annual Meeting of the American Institute of Chemical Engineers, Salt Lake City, UT, November 2007.

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4. Elabd, Y.A. Transport Phenomena in Polymer Electrolyte Membranes for the Direct Methanol Fuel Cell. NATO Advanced Study Institute on Mini-Micro Fuel Cells as Electric Energy Generators, Cesme, Izmir, Turkey, July 2007. Invited Speaker

5. Hallinan, D.T., Jr.; Elabd, Y.A. Transport of Water in Nafion® using Time-Resolved FTIR-ATR Spectroscopy. Annual Meeting of the North American Membrane Society, Orlando, FL, May 2007.

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8. Rahmathullah, A.M.; Elabd, Y.A.; Palmese, G.R. Structure, Property and Cure Kinetics of Thermosetting Copolymers. Annual Meeting of the American Institute of Chemical Engineers, San Francisco, CA, November 2006.

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17. Chen, H.; Palmese, G.R.; Elabd, Y.A. Membranes with Oriented Polyelectrolyte Nanodomains as Breathable Barriers for Protective Clothing. 25th Army Science Conference, Orlando, FL, November 2006.

18. Rahmathullah, A.M.; Elabd, Y.A.; Palmese, G.R. Plasma-Radiation Enhanced Nanofiber-Thermoplastic Composites. Annual Meeting of the American Institute of Chemical Engineers, San Francisco, CA, November 2006.

19. Hallinan, D.T., Jr.; Elabd, Y.A. Multicomponent Transport of Water and Methanol in Nafion®. Annual Meeting of the North American Membrane Society, Chicago, IL, May 2006. Received NAMS Poster Award ($500) and NAMS Annual Meeting Student Travel Award ($1,000)

20. Rahmathullah, A.M.; Elabd, Y.A.; Palmese, G.R. Multifunctional Nanoporous Thermosetting Copolymer Membranes: Synthesis and Characterization. Annual Meeting of the North American Membrane Society, Chicago, IL, May 2006. Received Elias Klein Founders' Travel Award ($500)

21. Rahmathullah, A.M.; Elabd, Y.A.; Palmese, G.R. Multifunctional Nanoporous Thermosetting Copolymers. American Chemical Society Local Section 6th Annual Poster Session, Villanova University, Philadelphia, PA, January 2006. Received the EssTech Polymers and Materials Science Award

22. Chen, H.; Rahmathullah, A.M.; Hallinan, D.T., Jr.; Napadensky, E.; Palmese, G.R.; Elabd, Y.A. Polymer-Polymer Nanocomposite Membranes as Breathable and Responsive Barriers. 2005 Scientific Conference on Chemical and Biological Defense Research, Timonium, MD, November 2005.

23. Rahmathullah, A.M.; Elabd, Y.A.; Palmese, G.R. Multifunctional Nanoporous Thermosetting Copolymer Matrices. American Society of Composites Annual Meeting, Philadelphia, PA, September 2005.

24. Rahmathullah, A.M.; Palmese, G.R.; Elabd, Y.A. Proton Conductive Nanoporous Thermosetting Copolymers, Fall National Meeting of the American Chemical Society, Washington, DC, August 2005.

Number of Presentations: 24.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

1. Chen, H.; Palmese, G.R.; Elabd, Y.A. Membranes with Oriented Polyelectrolyte Nanodomains as Breathable Barriers for Protective Clothing. 25th Army Science Conference Proceedings 2006, MP-01.

2. Chen, H.; Palmese, G.R.; Elabd, Y.A. Membranes with Oriented Polyelectrolyte Nanodomains. ACS Polymeric Materials: Science and Engineering 2006, 95, 269-270.

3. Chen, H.; Palmese, G.R.; Elabd, Y.A. Polyester-Poly(methacrylic acid) Nanocomposite Membranes as Breathable Barriers. ACS Polymer Preprints 2005, 46 (2), 1202-1203.

4. Mohamed Aflal, M.R.; Elabd, Y.A.; Palmese, G.R. Proton Conductive Nanoporous Thermosetting Copolymers. ACS Polymeric Materials: Science and Engineering 2005, 93, 564-565.

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts): 4

Peer-Reviewed Conference Proceeding publications (other than abstracts):

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts): 0

(d) Manuscripts

Number of Manuscripts: 0.00

Patents Submitted

1. Elabd, Y.A.; Palmese G.R. Filled Nanoporous Polymer Membrane Composites for Protective Clothing and Methods for Making Them. World Patent, filed May 24, 2007, international application number PCT/US07/69658, pending approval. U.S. Patent, filed November 17, 2008, application number 12/301,176, pending approval. Featured in Homeland Security Daily Wire, February 2008.

Patents Awarded

Awards

NSF CAREER Award (2007), DuPont Science and Engineering Award (2005)

Graduate Students

NAME	PERCENT_SUPPORTED
M. Aflal M. Rahmathullah	1.00
Daniel T. Hallinan, Jr.	0.25
Holly Schaeffer	0.25
FTE Equivalent:	**1.50**
Total Number:	**3**

Names of Post Doctorates

NAME	PERCENT_SUPPORTED
Hong Chen	1.00
Bin Dong	1.00
FTE Equivalent:	**2.00**
Total Number:	**2**

Names of Faculty Supported

NAME	PERCENT_SUPPORTED	National Academy Member
Yossef A. Elabd	0.08	No
Giuseppe R. Palmese	0.08	No
FTE Equivalent:	**0.16**	
Total Number:	**2**	

Names of Under Graduate students supported

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Student Metrics

This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period:	0.00
The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:.....	0.00
The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:.....	0.00
Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):	0.00
Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:	0.00
The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense	0.00
The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields:	0.00

Names of Personnel receiving masters degrees

NAME
Total Number:

Names of personnel receiving PHDs

NAME
M. Aflal M. Rahmathullah
Daniel T. Hallinan, Jr.
Total Number: 2

Names of other research staff

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Sub Contractors (DD882)

Inventions (DD882)

5 **Filled Nanoporous Polymer Membrane Composites for Protective Clothing and Methods for Making Them.**

Patent Filed in US? (5d-1) Y

Patent Filed in Foreign Countries? (5d-2) Y

Was the assignment forwarded to the contracting officer? (5e) Y

Foreign Countries of application (5g-2): World Patent

5a: Giuseppe Palmese

5f-1a: Drexel University

5f-c: 3141 Chestnut St

Philadelphia PA 19104

5a: Yossef A. Elabd

5f-1a: Drexel University

5f-c: 3141 Chestnut St

Philadelphia PA 19104

Scientific Progress

See Attachment

Technology Transfer

Final Report

Selective and Responsive Nanopore-Filled Membranes
PI: Y.A. Elabd
Co-PI: G.R. Palmese

The overall goal of this project is to develop a new class of breathable barrier membrane (i.e., selective for water vapor over harmful agents) with stimuli-responsive capabilities and the mechanical strength required for protective clothing for chemical and biological defense. These new materials are based on polymer-polymer nanocomposites of hydrophilic ionic polymer gels within a hydrophobic polymer host matrix. The specific tasks of this project include (1) synthesizing stimuli-responsive selective membranes based on polymer-polymer nanocomposites, (2) measuring transport properties: breathable barrier selectivity and electrical stimuli-responsive capabilities, and (3) measuring and modeling multicomponent transport properties and materials optimization. This work focuses on two new membrane concepts: (1) Nanopore-Filled Membranes and (2) Encapsulated Nanofiber Mesh Membranes. The goals of this project were accomplished, where the details of these results are documented in the annual reports and attached manuscripts.

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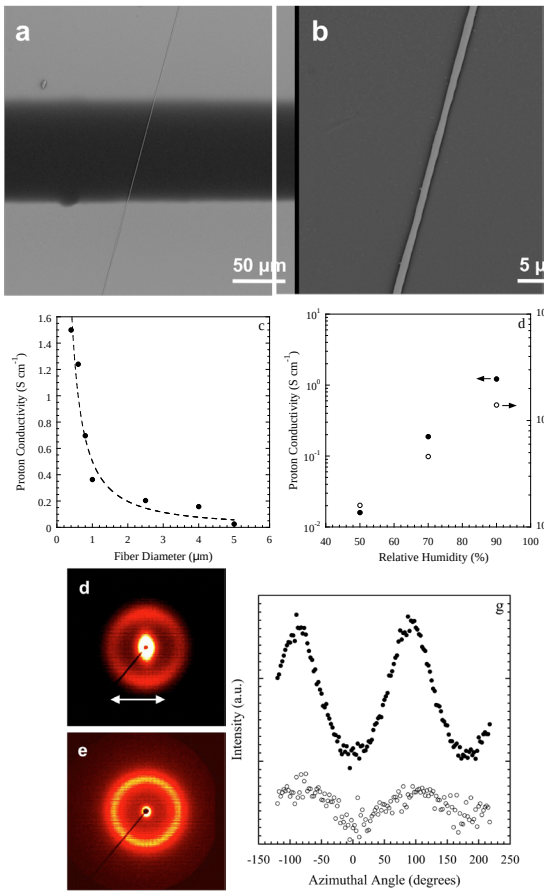

Figure 1. (a) SEM image of a single high purity Nafion nanofiber bridging two electrodes; (b) enlarged image of (a); (c) proton conductivity (at 30 ^{o}C, 90% RH) versus fiber diameter for high purity Nafion nanofibers measured on individual nanofibers; (d) Humidity-dependent proton conductivity (30 ^{o}C) of a single high purity Nafion nanofiber (solid symbols) and a cast Nafion film (open symbols); 2-D X-ray scattering patterns (at 86% RH, 25 ^{o}C) of (e) macroscopically aligned high purity Nafion nanofibers (arrows indicating longitudinal direction of fibers) and (f) Nafion cast film; (g) X-ray scattering intensity as a function of azimuthal angle for macroscopically aligned high purity Nafion nanofibers (solid symbols) and a cast Nafion film (open symbols).

bulk film. This work also demonstrates the successful fabrication of high purity Nafion nanofibers (~ 99.9 wt%) via electrospinning and higher humidity sensitivity for nanofibers compared to the bulk. These results should have a significant impact on not only the development of breathable barrier membranes, but also other applications such as fuel cells and sensors.

PUBLICATIONS

Publications *(Peer-Reviewed Archival Manuscripts)*

1. Dong, B.; Gwee, L.; Salas-de la Cruz, D.; Winey, K.I. Elabd, Y.A. Super Proton Conductive High Purity Nafion Nanofibers. *Nano Letters* **2010**, *10*, 3785-3790.

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9. Chen, H.; Palmese, G.R.; Elabd, Y.A. Membranes with Oriented Polyelectrolyte Nanodomains. *Chem. Mater.* **2006**, *18*, 4875-4881.

Publications *(Book Chapters)*

10. Chen, H.; Rahmathullah, A.M.; Palmese, G.R.; Elabd, Y.A. Polymer-Polymer Nanocomposite Membranes as Breathable Barriers with Electro-Sensitive Permeability; In *Nanoscience and Nanotechnology for Chemical and Biological Defense*. Nagarajan, R.; Zukas, W.; Hatton, T.A.; Lee, S., Eds.; Oxford University Press, 2009; ACS Symposium Series 1016, pp 307-322. **Invited Contribution**

11. Hallinan, D.T., Jr.; Elabd, Y.A. Sorption and Diffusion Selectivity of Methanol/Water Mixtures in Nafion®. In *Micro-Mini Fuel Cells – Fundamentals and Applications*; Kakac, S., Pramuanjaroenkij, A., Vasiliev, L., Eds; Springer: Netherlands, 2008; pp 189-208. **Invited Contribution**

Publications *(Archival Proceedings, Preprints, and Technical Reports)*

12. Chen, H.; Palmese, G.R.; Elabd, Y.A. Membranes with Oriented Polyelectrolyte Nanodomains as Breathable Barriers for Protective Clothing. *25th Army Science Conference Proceedings* **2006**, *MP-01*.

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PRESENTATIONS

Presentations *(Oral Presentations at Meetings and Conferences, presenter underlined)*

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21. Rahmathullah, A.M.; Elabd, Y.A.; Palmese, G.R. Multifunctional Nanoporous Thermosetting Copolymers. American Chemical Society Local Section 6th Annual Poster Session, Villanova University, Philadelphia, PA, January 2006. **Received the EssTech Polymers and Materials Science Award**

22. Chen, H.; Rahmathullah, A.M.; Hallinan, D.T., Jr.; Napadensky, E.; Palmese, G.R.; Elabd, Y.A. Polymer-Polymer Nanocomposite Membranes as Breathable and Responsive Barriers. 2005 Scientific Conference on Chemical and Biological Defense Research, Timonium, MD, November 2005.

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